Methylation of Metalation Product.—When the *n*-butyllithium metalation product of triphenylarsine was treated with methyl sulfate a 2.7% yield of *m*-tolyldiphenylarsine was obtained. Its mercuric chloride derivative, melting at 201°, was identical with that obtained from *m*-tolyldiphenylarsine described above.

DEPARTMENT OF CHEMISTRY
IOWA STATE COLLEGE
AMES, IOWA
RECEIVED OCTOBER 6, 1941

Hydrated Lithium Aluminum Sulfate (Lithium Alum)

BY HAROLD A. HORAN AND JOHN J. DUANE

In the course of additional work on the ternary system Li_2SO_4–$Al_2(SO_4)_3$–H_2O at 0° aimed at determining the degree of hydration of aluminum sulfate in equilibrium with saturated solutions, which will be reported later, an invariant point with solution composition differing from the previously reported one[1] has been found. Careful investigation of the region between these invariant points clearly shows the existence of a third phase in the system. Figure 1 is a photograph of this solid, its appearance being unlike that of either hydrated lithium or aluminum sulfates. The existence of lithium alum has been referred to before.[1,2,3] The present evidence clearly indicates the formation of a double salt of composition closely approximating $LiAl(SO_4)_2 \cdot 12H_2O$, which might be called lithium alum if the crystals are shown to have a structure similar to the other alums.

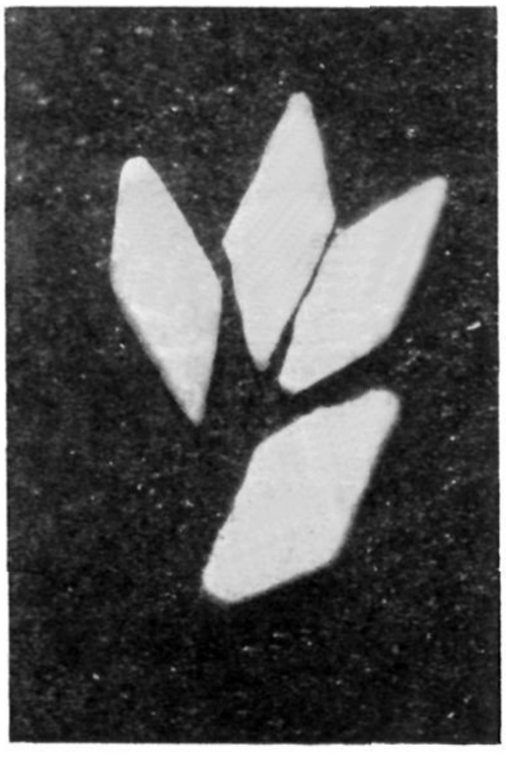

Fig. 1.—Hydrated lithium aluminum sulfate (×10).

Kralovansky, in 1828, may have found lithium alum, but many investigators since that time have been unable to repeat his work. The general opinion up to the present time is that lithium does not (and even cannot) form an alum.

Experimental

The methods used are essentially those of the previous investigation,[1] the only difference being the use of an analyzed concentrated solution of purified aluminum sulfate in making up the complexes. This locates the original complex with greater certainty. The wet residues are the corresponding solids, quickly separated from the solutions and centrifuged. The experimental data are given in Table I and the values are represented graphically in Fig. 2. The latter is only part of the isotherm, for obvious reasons.

TABLE I

PARTIAL ISOTHERM AT 0° FOR Li_2SO_4–$Al_2(SO_4)_3$–H_2O

Solution		Original complex		Wet residue		
Wt. % $Al_2(SO_4)_3$	Wt. % Li_2SO_4	Wt. % $Al_2(SO_4)_3$	Wt. % Li_2SO_4	Wt. % $Al_2(SO_4)_3$	Wt. % Li_2SO_4	Solid phase
16.66	14.65	17.96	18.98	...	...	I
16.63	14.59	20.06	17.01	...	...	I
17.23	14.11	22.45	13.68	34.75	12.60	II
17.31	13.94	23.38	13.59	33.82	12.66	II
17.85	13.28	23.96	13.10	37.65	12.47	II
18.05	13.02	22.11	12.52	...	...	III
18.07	13.07	22.62	12.16	...	...	III
18.05	13.08	23.91	11.18	...	...	III

The solid phases represented by the numerals I, II and III are respectively: $Li_2SO_4 \cdot 1H_2O + LiAl(SO_4)_2 \cdot 12H_2O$, $LiAl(SO_4)_2 \cdot 12H_2O$ and $LiAl(SO_4)_2 \cdot 12H_2O + Al_2(SO_4)_3 \cdot nH_2O$ (n not determined in this study).

Algebraic extrapolations[4] of the tie lines through the compositions of the saturated solutions, the original complexes and the wet residues to the theoretical $Al_2(SO_4)_3$ value in $LiAl(SO_4)_2 \cdot 12H_2O$, give Li_2SO_4 values all within 0.2% absolute of the theoretical Li_2SO_4 value demanded by this formula (point A, Fig. 2).

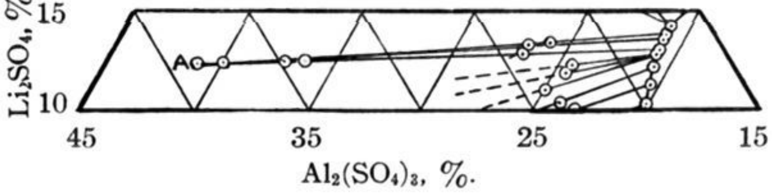

Fig. 2.—Partial isotherm 0°: Li_2SO_4-$Al_2(SO_4)_3$-H_2O.

Several samples of the unwashed solid, free from most of the adherent solution, were analyzed for

---

[1] Horan and Skarulis, THIS JOURNAL, **61**, 2689 (1939).

[2] Kralovansky, *J. für. Chem. und Physik*, **54**, 347 (1828).

[3] Rammelsberg, *Sitzgber. preus. Akad. Wiss., Berlin*, 385 (1848).

[4] Hill and Ricci, THIS JOURNAL, **53**, 4306 (1931).

aluminum and lithium. Typical results are: found: $Al_2(SO_4)_3$, 38.27, 38.36; Li_2SO_4, 12.87, 12.96. Calcd. for $LiAl(SO_4)_2 \cdot 12H_2O$: $Al_2(SO_4)_3$, 38.68; Li_2SO_4, 12.43.

The double salt is incongruently soluble at this temperature. This, and the limited region of existence, may explain the failure of many attempts during the last one hundred years to prepare this salt.

The authors are grateful to Professor John E. Ricci of New York University for helpful suggestions and to Dr. M. F. Reynolds of Queens College who assisted in obtaining the photograph shown in Fig. 1.

DEPARTMENT OF CHEMISTRY
ST. JOHN'S UNIVERSITY
BROOKLYN, N. Y.
RECEIVED SEPTEMBER 27, 1941

Preparation of Maltose Monohydrate by the Deacetylation of Maltose Octaacetate with Barium Methylate

BY WILLIAM A. MITCHELL

There has been some difficulty encountered in preparing pure maltose from the octaacetate following the procedure reported by Zemplén.[1] The use of the so-called barium methylate[2] as a deacetylating agent was found to work very well, however.

Preparation of Barium Methylate.—Slowly add 50 g. of barium monoxide to 150 cc. of absolute methyl alcohol. After the initial reaction has subsided, gently reflux for one hour, dilute to 300 cc., and filter. Standardize with approximately normal sulfuric acid using phenolphthalein indicator and store in refrigerator.

The Deacetylation of the Octaacetate[3] of Maltose.—Fifty grams of the octaacetate is dissolved in 500 cc. of absolute methyl alcohol. The solution is cooled to about 10°, and 10 cc. of barium methylate solution added while the flask is well agitated. After an hour or more of intermittent shaking, the flask is cooled in an ice-bath and an exact equivalent of the standard sulfuric acid slowly added. The barium sulfate is allowed to settle overnight and the supernatant liquid decanted. To facilitate the removal of the almost colloidal barium sulfate, the suspension is heated in a water-bath for several minutes with 5 g. of activated carbon plus 5 g. of analytical grade filter-cel. This mixture is filtered until a clear solution is obtained. The water-white solution is concentrated over a steam-bath using a good vacuum. The drying is complete when the material in the flask is snow white and able to be powdered when prodded with a stirring rod.

Crystallization of Maltose Monohydrate.—The above powdered maltose is weighed quickly and 0.4 cc. of water added for each gram of dry maltose. This mixture is heated in hot water until a thick sirup is formed. Now 65 cc. of 95% ethyl alcohol is added, the solution is heated for several minutes with 2 g. of carbon and 2 g. of filter-cel, and then filtered. The residue may be washed with a small amount of hot 80% alcohol. The clear filtrate is allowed to cool and then seeded with a small crystal of maltose and allowed to crystallize at room temperature with the aid of mechanical stirring. The crystallization is usually complete in about three days, but in order to ensure maximum yield, the maltose should be allowed to stand in the refrigerator for an additional three or four days before filtering and washing with 95% alcohol. The crystals are dried in a vacuum oven at 45° for two days. The yield is 50–60% of the theoretical amount. The specific rotations of five different preparations made by the above procedure varied over the range $[\alpha]^{26}\mathrm{D}$ +129.5 to 130.0°.

To compare this maltose with the best commercial preparation and maltose recrystallized from water–alcohol solutions, the reducing power was measured using the method of Blish and Sandstedt.[4]

NUMBER OF CC. OF 0.1 N POTASSIUM FERRICYANIDE REDUCED BY DIFFERENT PREPARATIONS OF MALTOSE

	Sample of maltose			
Maltose used, mg.	Best grade maltose obtained commercially	Good grade maltose recrystallized several times	Sample obtained from another laboratory—considered pure	Maltose prepared through the octaacetate
10.0	3.58	3.60	3.60	3.69
15.4	5.29	5.35	5.34	5.40
20.0	6.76	6.90	6.86	6.87
25.0	8.28	8.40	8.36	8.44

(After the method of Blish and Sandstedt, *Cereal Chem.*, **10**, 189 (1933)).

It is apparent that the reducing values of the different preparations are very close to each other.

CENTRAL LABORATORIES
GENERAL FOODS CORPORATION
HOBOKEN, N. J.
RECEIVED OCTOBER 2, 1941

---

[1] Zemplén, *Ber.*, **59**, 1258 (1926); Zemplén, *ibid.*, **60**, [2] 1555 (1927); Zemplén and Pacsu, *ibid.*, **62**, 1613 (1929).

[2] Weltzien and Singer, *Ann.*, **443**, 104 (1925); Isbell, *Bur. Standards J. Research*, **5**, 1185 (1930).

[3] The yield of maltose octaacetate can be increased considerably by reclaiming the solid from the alcohol mother liquors and again treating with a portion of acetic anhydride and sodium acetate.

[4] Blish and Sandstedt, *Cereal Chem.*, **10**, 189 (1933).

Invert Soaps of Naphthalene

BY JOSEPH B. NIEDERL AND HERSH WEINGARTEN[1,2,3]

It was shown in the work by G. Domagk[4] and R. Kuhn and co-workers[5] that invert soaps pos-

---

[1] This communication was part of a paper presented before the Division of Medicinal Chemistry at the Atlantic City Meeting of the American Chemical Society; Abstracts of Papers, 102nd Meeting, American Chemical Society, Atlantic City, New Jersey, September 8–12, 1941, pp. K-8.

[2] Abstracted from the thesis presented by H. Weingarten to the Graduate School of New York University in partial fulfillment of the requirements for the degree of Master of Science, June, 1942.

[3] J. B. Niederl and co-workers, THIS JOURNAL, **63**, 945, 1475, 1476, 2024 (1941).

[4] G. Domagk, *Dtsch. med. Wschr.*, **I**, 829 (1935).

[5] R. Kuhn and co-workers, *Ber.*, **73**, 1080–1109 (1940).